UNITED STATES PATENT OFFICE

2,690,964

PROCESS FOR GELLING LIQUID NITROPARAFFINS

Herman Maisner, Los Angeles, Calif., assignor, by mesne assignments, to Aerojet-General Cor-poration, Cincinnati, Ohio, a corporation of Ohio

Original application February 1, No Drawing. 1947, Serial No. 725,977. Divided and this application February 23, 1952, Serial No. 273,779

3 Claims. (Cl. 52-13)

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This invention relates to nitroparaffin-containing substances, and has for its principal object to provide a gel containing nitroparaffin, and a process of forming it.

This is a division of my copending application 5 Serial No. 725,977, filed February 1, 1947.

Liquid nitroparaffins have heretofore been employed as propellants in jet propulsion devices, and they have also been used in explosives. It is desirable for some types of operating conditions 10 to employ the liquid nitroparaffins in a solid or semi-solid form, or gel, for these purposes. Up to the present time, difficulty has been encountered when it was attempted to produce gels from the various liquid nitroparaffins without seriously 15 affecting their oxygen balance and otherwise changing their properties.

By the present invention I have discovered a method for thickening, gelling, or solidifying liquid nitroparaffins and forming semi-solid or solid 20 gels which retain their consistency for a period of time without undergoing any marked change.

This invention is, in general, directed to solidifying or gelling any of the liquid nitroparaffins including the mono, and poly nitromethanes, 25 mononitroethane, mononitropropane, mononitroisopropane, mononitrobutanes, mononitroisobutanes, mononitropentanes and the like which are normally liquid compounds at room temperatures. Thus, I may employ any of the liquid nitroparaffins, such as mononitromethane (commonly called nitromethane, and so designated hereinafter), dinitromethane, nitroethane, nitropropane, nitrobutane, and the like or any mixtures of the liquid nitroparaffins or solutions of any of the normally solid polynitroparaffins having sufficient oxygen to sustain self-combustion in nitromethane. Nitromethane is usually the preferred nitroparaffin for propellant and explosive purposes, in that it forms a very satisfactory nitrocellulose and employed in the manner in which such propellants are usually used; and a very satisfactory explosive when gelled in combination with a sensitizer or otherwise treated and handled in the manner of an explosive.

I have discovered that it is possible to produce a homogeneous semi-solid or solid gel from liquid nitroparaffins by dissolving or adding to the liquid nitroparaffins, such as nitromethane, nitroethane and the like, a quantity of nitrocellulose. 50 Various commercial forms in which nitrocellulose suitable for the purpose, may be obtained are: Pyrocellulose, pyroxylin, guncotton, etc., all of which are trade names for such nitrocelluloses.

The gelling of the nitroparaffins is preferably conducted at room temperatures, or temperatures not more than slightly above room temperatures, since both nitroparaffin and nitrocellulose become explosive when confined and subjected to more elevated pressures and temperatures. This is particularly true when a sensitizing agent has been added to nitromethane to assure its action as an explosive. At elevated temperatures, that is, above 350° F., nitromethane, for example, becomes extremely sensitive to shocks and at still higher temperatures may explode without subjecting the liquid to any shock whatsoever.

The gels produced from nitrocellulose and a liquid nitroparaffin may be compounded, by adjusting the proportions to give gels having varying degrees of viscosity. These range from syrupy materials to solid gels. For practical purposes the gels employed vary from between about 5% to 75% nitrocellulose by weight based on the weight of nitroparaffin used. Gels may be made to contain any amount above 75% nitrocellulose by weight based on the weight of the total mixture, but even 75% nitrocellulose gels are practically solid, therefore, there is little advantage from this standpoint in using a higher percentage of nitrocellulose.

Since nitroparaffins of the type of nitromethane, and nitrocellulose both have sufficient oxygen in their molecules to insure their complete combustion, these substances are capable of burning to completion at ambient conditions without requiring the addition of oxidizers. Gelled nitroparaffins such as nitromethane are particularly useful where it is desired to employ these substances as solid or semi-solid explosives or as self-combustible solid propellant fuels such as are used in self-contained rocket motors.

The nitroparaffin gels prepared according to self-combustible propellant when gelled with 40 my invention may be employed either as explosives or as self-combustible propellants, depending upon the manner of use. The gels are explosive when detonated with sufficient force but may readily be adapted to be burned as fuels, 45 particularly in jet propulsion motors, in the absence of detonation. If it is desired to prepare a nitroparaffin gel to be used as an explosive I may incorporate in the liquid nitroparaffin, prior to gelling, a sensitizer. Such sensitizers as the organic amines disclosed by Laurence in patent application Ser. No. 605,747, filed July 18, 1945, are highly satisfactory, including the aromatic amines such as aniline, diphenylethylenediamine and the like and the aliphatic amines such as 55 diethylamine, tetraethylene pentamine and the

like. A nitroparaffin gel in which a sensitizer such as one of the organic amines is incorporated is much more sensitive to detonation than is the gel in the absence of the sensitizer, although it is to be understood that the use of a sensitizer is optional; and the gels may be employed as explosives either with or without a sensitizer.

Thus mixtures of nitromethane or other nitroparaffins and nitrocellulose containing from 10 about 75% to about 85% by weight of nitroparaffin based on the total weight may be satisfactorily used as explosives in conjunction with a suitable detonator. Higher proportions of nitrocellulose may be employed; this tends to de- 15 crease the explosive strength.

Mixtures of nitroparaffin and nitrocellulose containing from 79% to 83% by weight of nitroparaffin such as nitromethane based on the total weight of the mixture are easily exploded 20 by the shock of a No. 8 fulminate type of blasting cap, yet in the absence of shock, such mixtures (containing as high as 74% to 84% by weight of nitromethane in combination with factorily as restricted propellant charges without exploding. A restricted propellant charge is defined as one in which the burning occurs over a predetermined face of limited area and does not take place simultaneously over the en- 30 tire outside surface of the propellant mass. Restriction may be accomplished by coating all surfaces except the burning surface with a liner which is adhesive to the propellant mass and forming the principal charge. Examples of adhesive liners suitable for confining the burning are ethylcellulose, cellulose acetate, cellulose butyrate, cellulose acetate butyrate, etc.

The burning properties of solid propellant 40 charges compounded by gelling nitromethane with nitrocellulose may be greatly improved by adding suitable catalysts to the nitromethane before gelling. A particularly desirable catalyst for assisting the easy ignition of nitromethane is chrome acetyl acetonate. Small amounts of this substance ranging between a trace up to approximately 3% by weight may be used to improve the burning of nitromethane.

The ratio of nitroparation to nitrocellulose 50 may, as stated, be varied over a wide range of proportions ranging from a trace of nitrocellulose in liquid nitropraffin to a trace of nitroparaffin in nitrocellulose. The amount of nitrocellulose which may be incorporated into the liquid nitroparaffin by stirring or agitation alone will not appreciably exceed 45% based on the weight of the nitroparaffin employed.

A fairly substantial gel may be formed by dissolving in nitroparaffin as low as 10% nitrocellulose by weight based on the weight of the nitroparaffin while a reasonably stiff gel is obtained when the amount of nitrocellulose employed is increased to 20% or 30% by weight based on the weight of the nitroparaffin employed. Particularly stiff gels are formed when 40% to 50% by weight of nitrocellulose based on the weight of nitroparaffin present is employed.

If it is desired to incorporate amounts of nitrocellulose in excess of 45% by weight, it is $_{70}$ preferable to mill or knead the mixtures of nitrocellulose and a liquid nitroparaffin to insure a more homogeneous mixture.

The process herein described possesses the ad-

be easily produced from nitroparaffins. Sensitized nitroparaffin such as nitromethane may, by careful selection of the proportions of nitromethane to nitrocellulose, be used either as a solid or semi-solid explosive. Non-sensitized nitromethane and other self-combustible nitroparaffins may be made into self- combustible solid propellants, or may be used as explosives when detonated with sufficient force.

The following examples of gels made according to my invention serve to illustrate the application of the invention.

Example 1.—A gel consisting of 80% by weight of nitromethane and 20% by weight of nitrocellulose has been prepared by dissolving the nitrocellulose in the nitromethane at 75° F. The gel thus formed was very stable and fairly rigid. Additional gels consisting of nitromethane and nitrocellulose were made in similar manner and contained nitrocellulose varying in proportions from about 10% to 50%.

Example 2.—Numerous other gels have been prepared by dissolving nitrocellulose in nitropropane and nitroethane at temperatures rangnitrocellulose) may be confined and burned satis- 25 ing from approximately 70° F. to 90° F. These gels consisted of from about 50% to 90% by weight of the respective nitroparaffin and from about 10% to 50% by weight of nitrocellulose.

Example 3.—A gel consisting of 14.6% by weight of nitrocellulose dissolved in 85.4% of nitromethane formed a thick fluid gel.

Example 4.-- A gel consisting of 25% nitrocellulose and 75% nitromethane was prepared by dissolving the nitrocellulose in the nitromethane has a slower burning rate than the material 35 at approximately 75° F. This produced a fairly stiff gel.

Example 5.-A solution of 50% nitromethane into which has been dissolved 50% of nitrocellulose produced a very stiff gel having substantially solid properties.

Example 6.—A sample of a gel consisting of 75.3% nitromethane which had dissolved therein 22.9% nitrocellulose was made more active by the addition of 1.8% of chrome acetyl acetonate. This formed a fairly stiff gel having suitable burning properties for a restricted propellant charge.

Example 7.—29.8% of nitrocellulose was dissolved in 69.2% of nitromethane and 1.0% of chrome acetyl acetonate was added to the mixture. This formed a stiff gel which appears to be very satisfactory when employed as a solid propellant charge.

Other gels which may be satisfactorily formed due to the fact that nitrocellulose is a good gelling agent for nitromethane and similar nitroparaffins are those which are made from solutions of solid organic polynitrocompounds in nitromethane. Examples of these substances are trinitrotoluene, polynitrobenzene, dinitropropane, dinitroethane and other higher polynitroparaffins which possess sufficient oxygen in their molecules to sustain their self-combustion. These are selected to produce propellants having the required properties such as hardness, temperature coefficient and freedom from detonation.

An additional advantage of my process apparent from the foregoing examples, is that the ingredients may be compounded at ambient temperatures and require little or no heating, thus eliminating the hazard present when propellants are subjected to temperatures greatly in excess of ambient. This is particularly desirable in vantage that any desired consistency of gel may 75 view of the fact that nitro-paraffins, such as Mumbar

A particularly desirable feature, inherent in restricted burning propellant charges formed from solid gels of the above ingredients, is that the gases produced when these charges are burned in a jet motor are substantially smokeless, noncorrosive and nontoxic. The specific impulse obtained by burning nitro-methane, or any other of the nitroparaffins having sufficient oxygen in the 10 molecule to support their own combustion, in combination with nitrocellulose is not appreciably lower than that developed by the nitroparaffin alone.

Gels methane and nitrocellulose may be formed into sticks similar to dynamite and may be substituted for dynamite since sensitized nitromethane when exploded compares favorably with nitroglycerin in shattering properties and power. The addition of nitrocellulose does not appear to affect the ease with which sensitized nitromethane can be exploded when subjected to shocks.

I claim:

1. A composition of matter comprising from 2 about 10% to 50% by weight of nitrocellulose, 50% to 90% by weight of nitromethane, and a sensitizer from the group consisting of aniline, diphenylethylenediamine, diethylamine and tetraethylene pentamine.

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2. A composition of matter consisting of a mixture of a liquid nitroparaffin, nitrocellulose and a sensitizer consisting of organic amine from the group consisting of aniline, diphenylethylenediamine, diethylamine and tetraethylene pentamine, the weight of the nitroparaffin being between 50% and 85% by weight based on the weight of the total mixture, and the weight of the nitrocellulose being between 15% and 50% by weight based on the weight of the total mixture.

3. A composition of matter consisting of a nitromethane, nitrocellulose and a sensitizer comprising an organic amine from the group consisting of aniline, diphenylethylenediamine, dicompounded from sensitized nitro- 15 ethylamine and tetraethylene pentamine, the weight of the nitromethane being between 50% and 85% by weight based on the weight of the total mixture, and the weight of the nitrocellulose being between 15% and 50% by weight based on 20 the weight of the total mixture.

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